

Technical Report

Experimental investigation of the low speed impact characteristics of nanocomposites

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ABSTRACT

Nanocomposites have attracted the attention of scientists during the past few decades due to their superior mechanical, thermal, chemical and electrical characteristics. This paper presents the potential of using nanoclay woven Kevlar laminated composites to enhance the impact energy resistance and mechanical performance. The variation of nanoclay percentage usually leads to different characteristics of the resulting composite, and an increase in energy absorption by high percentage of fillers may accompany tendency to delamination. The effect of different percentages of nanoclays on composite properties are investigated in this paper. The results revealed enhancement in delamination resistance at low percentages of nanoclay additives, while the high percentage of nanoclay aggravated the composite delamination. The nanoclay with low percentage (4.3 wt.%) had shown the best results in delamination resistance.

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1. Introduction

The widespread use of nanocomposites attracted the attention of scientists in many engineering applications due to their superior mechanical, thermal, chemical and electrical characteristics. The introduction of inorganic nanoparticles as additives into polymer systems has resulted in polymer nanocomposites exhibiting multifunctional, high-performance polymer characteristics beyond what traditional filled polymeric materials possess. The development of these new materials enables the circumvention of classic material performance trade-offs by accessing new properties and exploiting unique synergies between materials, that only occur when the length scale of morphology and the fundamental physics associated with a property coincide, i.e., on the nano scale level. Through control/alteration of the additives at the nano scale level, one is able to maximize property enhancement of selected polymer systems to meet or exceed the requirements of current military, aerospace, and commercial applications. The technical approach involves the incorporation of nano particles into selected polymer matrix systems whereby nano particles may be surface-treated to provide hydrophobic characteristics and enhanced inclusion into the hydrophobic polymer matrix [1,2].

One of the important characteristics of nanocomposites that lead to their wide use in many industrial applications is their improved impact resistance. Woven fabric laminates have proved to

have superior impact energy absorbing properties to those of laminates made of unidirectional prepreps [3]. These fabrics are used in a number of engineering applications across various industries, including such products as automobile airbags; flexible structures like boat sails and parachutes; reinforcement in composites; architectural expressions in building roof structures; protective vests for military, police, and other security circles; and protective layers around the body in planes.

Woven fabrics consist of yarns woven in the fill and the warp directions. In general, such fabrics exhibit a significant stiffness only along the yarn directions under tension. The tensile response in the fill and warp directions may be coupled due to the crimp exchange mentioned above. Woven fabrics typically have negligible stiffness in bending and in-plane compression [4].

The main objective of the current study is to investigate the potential use of nanoclay particles in woven Kevlar laminated composites to enhance the composite impact energy resistance and mechanical properties. The variation of nanoclay percentage usually leads to different characteristics of the resulting composite, and an increase the energy absorption by high percentage of fillers may accompany tendency to delamination. To investigate the effect of nanoclay particles addition, different percentages of nanoclays were utilized. The results were compared to control samples composed of Kevlar plies only. Many researchers have studied the impact characteristics of nanoclay composites, in which they have utilized different polymer and fiber materials [5–9].

The laminated composites were prepared manually of fifteen plies of woven Kevlar 49 arranged in symmetrical 0/45 alternation.

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The samples were prepared by painting the matrix mix over each ply using painting brushes, then rolling each ply using a metallic roller to insure saturation of the resin and complete bonding between layers. After that, composite hot pressing technique is performed to insure plies perfect bonding and unified thickness and to expedite the curing.

An in-house developed vertical drop-weight experimental set-up was utilized to test the prepared sample laminates. This setup was built according to the guidelines given in the standard specifications ASTM: D7136. The test procedure determines the damage resistance of multidirectional polymer matrix composite laminated plates subjected to a drop-weight impact event. The composite material forms are limited to continuous-fiber reinforced polymer matrix composites. A flat rectangular composite plate is subjected to an out-of-plane, concentrated impact using a drop-weight device with a cylindrical impactor. The potential energy of the drop-weight is specified prior to each test. The damage resistance is quantified in terms of the resulting size and type of damage in the sample. The damage resistance properties generated by this test method are highly dependent upon several factors, which include specimen geometry, layup, impactor geometry, impactor mass, impact force, impact energy, and boundary conditions. Thus, the results are generally not scalable to other configurations, and are particular to the combination of geometric and physical conditions tested.

2. Materials used in preparing samples

The control samples are composite lamina prepared by adding 56 g of vinylester to 117 g of Kevlar 49, thus making a resin percentage of 32. The nanoclay composite test samples are prepared by adding nano montmorillonite clay, nanomer 1.34TCN that contains 25–30 wt.% to the vinylester in different percentages. Nanoclay is the most widely investigated nanoparticle in a variety of different polymer matrices for a spectrum of applications [10]. The origin of bentonite (natural clay) is most commonly formed by the in situ alteration of volcanic ash. Nanoclays have become attractive materials because of their potential use in wide range of applications such as in polymer nanocomposites [11]. Two different percentages of Nanoclay Kevlar composites were prepared in this work; namely, 4.3%, and 9.4%.

Kevlar is an aramid fiber, a term invented as an abbreviation for aromatic polyamide developed in 1965 by DuPont Company. The chemical composition of Kevlar is poly para-phenyleneterephthalamide, and it is more properly known as a para-aramid. The aramid ring gives Kevlar thermal stability, while the para structure gives it high strength and modulus [12]. Kevlar aramid has a high tensile strength, higher tensile modulus and lower density than fiber glass but it is more expensive than glass fiber [13]. Aramid fibers provide the highest tensile strength-to-weight ratio among reinforcing fibers. They provide good impact strength and, like carbon fibers, provide a negative coefficient of thermal expansion.

3. Sample preparation

3.1. Control samples

The control samples were prepared by mounting fifteen woven Kevlar 49 layers (17 × 17 cm size and 0.125 mm thickness) arranged symmetrically in 0°/45° orientations. The Kevlar plies were bonded together with vinylester resin. K-12 hardener with 0.5 wt.% was added to insure and accelerate the vinylester curing. The percentage of vinylester was determined by the minimum amount of resin required to insure complete saturation of Kevlar layers by resin. The fiber and matrix percentages were calculated after the

complete curing of the composite. The total weight of Kevlar layers was measured and recorded before composite samples preparation. The total weight of the composite after resin curing was measured and the weight of matrix phase is the difference between the composite and the weight of fiber.

The desired composite thickness was built up by placing various layers of the fiber and resin mixture. Consolidation of continuous fiber composites involves two important processes: resin flow through porous media and elastic fiber deformation [14,15]. There are various consolidation models that ignore the fiber deformation and consider only resin flow [16]. Heat was supplied during processing to expedite the cure rate of the resin.

Regular painting brush was utilized to distribute the resin evenly over the first Kevlar ply; the second ply was placed in 45° angle over the first ply (0° angle). Consolidating metallic roller was scrolled over the second ply to force the resin to penetrate the second ply, eliminate air bubbles inside resin and to eliminate cavities between the two Kevlar layers. After that resin was brushed again over the second ply and the third Kevlar ply was laid down in 0° orientation (the same orientation of the first Kevlar ply). The same scenario was adapted to prepare the rest of Kevlar layers. After that, the specimen was placed inside the hot press machine, the two pressing plates were sprayed by mold release agent and heated up to 175 °C, then the laminated composite was compressed by a force of 4500 N distributed over 30 × 30 cm area for 15 min. The composite lamina (15 layers of Kevlar + vinylester) was prepared by adding 56 g of vinylester to 117 g (15 layers) of Kevlar. The resin percentage was 32 wt.% of the composite and the Kevlar fibers 68 wt.%.

3.2. Nanoclay (NC) composite samples

Nanoclay Montmorillonite clay, nanomer 1.34TCN (Sigma–Aldrich, Missouri, USA) that contains 25–30 wt.% methyl dihydroxyethyl hydrogenated tallow ammonium was added in different percentages to the vinylester to manufacture different sets of samples. Two different percentages of Nanoclay Kevlar composites were prepared, 4.3%, and 9.4%. The same percentage of K-12 hardener was used (0.5 wt.%), and the samples were cured at room temperature for complete four days and then heated at 120 °C for 60 min.

4. Experimental setup

The impact energy absorption characteristics of the nanoclay Kevlar composites were investigated using an impact energy test. This test was carried out by utilizing a vertical drop weight experimental set up with a tube or rails to guide the falling weight during the free fall with known height and weight. This setup was built according to guidelines given in the ASTM: D7136. The setup, which was originally developed for testing rigid plastics, is shown schematically in Fig. 1. The main advantages of such set up is that it is unidirectional with no preferential direction for failure, can be used to test molded samples and samples do not have to shatter to be considered failures. These factors make falling weight testing a better simulation of functional impact exposures, and therefore closer to real-life conditions.

The developed setup is appropriate to test samples with 50 mm × 50 mm square dimensions, and 1–10 mm thick. Each tested sample was supported over a hollow steel chamber with an inside dimension of 50 × 50 × 10 mm. The cylindrical steel striker has 25.4 mm diameter and 50 mm length, and is allowed to fall from a height of up to 1.3 m onto the sample, which corresponds approximately to a potential energy of 380 J. This energy amount is considered as the maximum capacity of the setup. For a puncture

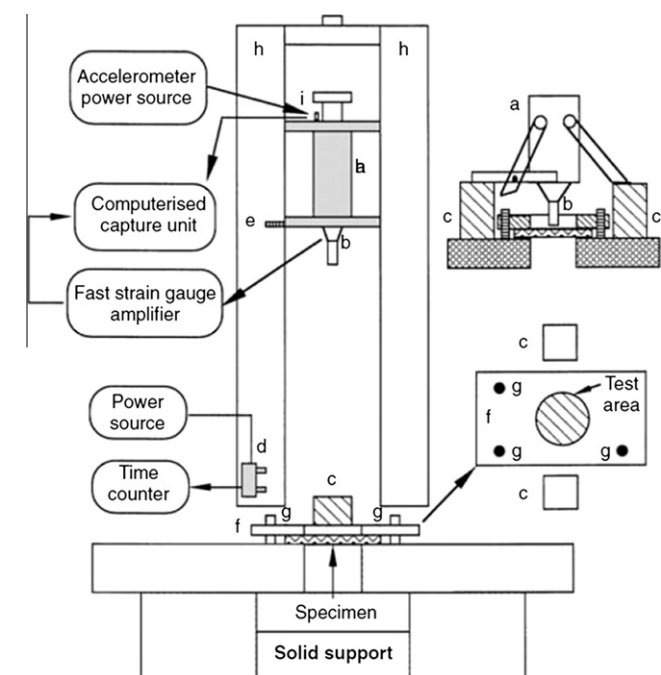


Fig. 1. An experimental setup for drop-weight impact tests (a) impactor, (b) strain-gauged load cell, (c) rebound catch block, (d) photodiodes, (e) flag, (f) clamping device, (g) locking pin, (h) drop guide, (i) accelerometer.

test on a composite, a strike velocity of 4.4 m/s and a total striker mass of 50–200 N (equivalent to energies of 48–193 J) are preferred [17].

The test method adopted in this research determines the damage resistance of multidirectional polymer matrix composite laminated plates subjected to a drop-weight impact event. The composite material forms are limited to continuous-fiber reinforced polymer matrix composites. A flat, rectangular composite plate is subjected to an out-of-plane, concentrated impact using a drop-weight device with a cylindrical impactor. The potential energy of the drop-weight, as defined by the mass and drop height of the impactor, is specified prior to each test. The damage resistance is quantified in terms of the resulting size and type of damage in the specimen. The damage resistance properties generated by this test method are highly dependent upon several factors including specimen geometry, layup, impactor geometry, impactor mass, impact force, impact energy, and boundary conditions. Thus, results are generally not scalable to other configurations, and are particular to the combination of geometric and physical conditions tested.

The samples were simply supported inside the clamping device, locking pin was fastened to prevent sample from sliding during impact, the weight lifted manually by rolling handle, the guiding tower tubes were lubricated to insure minimum friction, then the height measured and recorded, the pin removed and the weight free fell over the specimen. Similar procedures were adopted by Hallett and Ruiz [18] to study the response of unidirectional reinforced carbon/epoxy beams consisting of alternate 0°/90° plies under low speed impact.

After samples were prepared, they were tested under a low speed impact using the above described setup. The weight was lifted manually by the rolling handle to the desired level, the sample was placed between the fixing plates (specimen chamber), then the locking screw was placed to prevent any lateral sliding, after that the weight was released toward the sample. After the impact, the weight was lifted again to release the impacted sample, the locking screw was removed, and the sample was liberated. Each test trial, each test was followed by extensive visual investigation

to capture any failure happened. The number of removed layers and the delamination between the woven Kevlar layers in the lateral direction was noticed and recorded.

The control samples were used to evaluate the minimum energy required to completely penetrate the composite specimens. This amount of energy is considered to be a constant parameter during the entire research. The different composition composites were impacted with the same amount of energy. The height was varied and the failure mode was noticed. complete penetration took place at 85 cm and above heights. With a load mass of 30 kg, this is equivalent to 250 J potential energy.

5. Low speed impact test results and discussion

All prepared samples were of 50 mm × 50 mm size with thickness varying between 3.10 and 5.65 mm. Fourteen control samples were tested which were divided into two groups (DW1 and DW2). Group DW1 contains nine samples while group DW2 contains five samples. Each of the control samples is composed of 15 woven Kevlar layers with fiber fraction equal 73 wt.%. Seven nanoclay composite samples divided into two groups were also tested. The first group, DW5, contains three samples, each has NC of 4.30%. The second group, DW6, contains four samples, each has NC of 9.40%. Table 1 shows the description of the four groups of samples with their corresponding fiber and matrix weights and additive percentage.

Samples of group DW1, as shown in Table 2, showed considerable variation in thickness which is due to the manual preparation process and inclination of one of the hot press machine plates. To eliminate this variation, the composite samples of group DW2 was placed between two aluminum plates when pressed by the hot press machine. As a result, the samples of this group showed homogeneity in thickness.

The control samples of groups DW1 and DW2 were tested under different impact energies to predict the minimum energy required to completely penetrate the sample. The delamination and fiber fracture modes in the samples of group DW2 were similar to those of group DW1. The results which are shown in Tables 2 and 3 demonstrate that the complete penetration energy threshold is 250 J. The subsequent tests of the nanoclay composite samples were done at this amount of energy. Fixing the amount of test energy is necessary to examine the effect of adding different percentage of nanoclay to the composite matrix. In addition to the fiber fracture and matrix cracking, delamination was noticed in all tested samples. Delamination is one of the most common types of damage in laminated fiber reinforced composites due to their relatively weak interlaminar strength.

The Nanoclay samples of group DW5 contained 4.30 wt.% of NC. This percentage of NC was enough to increase the penetration resistance of the composite and only partially penetration of the composite samples was observed in all tests. The results of the visual inspection of the impacted samples are shown in Table 4 which did not show any evidence of delamination.

Group DW6 of the second nanoclay reinforced composites contained 9.40 wt.% of NC. As shown in Table 5, this percentage enhanced the impact resistance of the samples in a noticeable way and the impactor was not able to penetrate the composite samples in all four tests. Table 5 also demonstrates that the impacted samples showed high tendency of the samples to delaminate and showed almost complete delamination in some samples. It can also be stated that no delamination is observed with the 4.30% NC composite. Improvement in energy absorption can be attributed to the addition of nanoclay which led to improvement in the interlaminar shear strength as reported by Esfahani and Iqbal [3,19]. The addition of nanoclay increases delamination resistance of polymer

Table 1

Coding system used to represent the tested samples.

Sample type	Sample no.	Composition	Fiber weight (g)	Matrix weight (g)	Additive in wt.% of composite
Control	DW1	15 Kevlar layers + vinylester	78	37	0.00
	DW2	15 Kevlar layers + vinylester	78	29	0.00
Nanoclay	DW5	15 Kevlar layers + vinylester + NC	78	59	4.30
	DW6	15 Kevlar layers + vinylester + NC	78	132	9.40

Table 2

Results of the control sample group DW1.

Sample	Thickness (mm)	Height (cm)	Impact energy (J)	Response
DW1-1	3.25	115	338	Complete penetration + delamination ^a
DW1-2	3.30	111	327	Complete penetration + delamination ^a
DW1-3	3.40	110	324	Complete penetration + delamination ^a
DW1-4	3.15	110	324	Complete penetration + delamination ^a
DW1-5	3.30	104	306	Complete penetration + delamination ^a
DW1-6	3.15	94	277	Complete penetration + delamination ^a
DW1-7	3.50	94	277	Complete Penetration
DW1-8	3.50	85	250	Complete penetration + delamination ^a
DW1-9	3.30	75	221	Partial penetration + delamination ^a

^a Delamination was noticed by visual inspection.**Table 3**

Results of the control Samples DW2.

Sample	Thickness (mm)	Height (cm)	Impact energy(J)	Response
DW2-1	3.10	89	262	Complete penetration + delamination ^a
DW2-2	3.10	87	256	Complete penetration + delamination ^a
DW2-3	3.10	86	253	Complete penetration + delamination ^a
DW2-4	3.10	85	250	Complete penetration + delamination ^a
DW2-5	3.05	83	244	Partial penetration (two plies remain) + delamination ^a

^a Delamination was noticed by visual inspection.**Table 4**

Results of the nanoclay samples DW5 (4.30 wt.%).

Sample	Thickness (mm)	Height (cm)	Impact energy (J)	Response
DW5-1	4.50	85	250	Partial penetration (5 plies removed) + no delamination ^a
DW5-2	4.55	85	250	Partial penetration (5 plies removed) + no delamination ^a
DW5-3	4.65	85	250	Partial penetration (5 plies removed) + no delamination ^a

^a Delamination was noticed by visual inspection.**Table 5**

Results of the nanoclay samples DW6 (9.40 wt.%).

Sample	Thickness (mm)	Height (cm)	Impact energy (J)	Response
DW6-1	5.65	85	250	No penetration (2 plies removed only) + almost complete delamination at the mid of thickness ^a
DW6-2	5.25	85	250	No penetration (2 plies removed only) + almost complete delamination at the mid of thickness ^a
DW6-3	5.25	85	250	No Penetration (2 plies removed only) + almost complete delamination at the mid of thickness ^a
DW6-4	5.65	85	250	No penetration (2 plies removed only) + almost complete delamination at the mid of thickness ^a

^a Delamination was noticed by visual inspection.

composite, a dominating mechanism in absorption energy of polymer composite laminates during low and high-velocity impact tests, especially, in thick laminates [3]. Furthermore, specimens with 4.3 wt.% nanoclay showed improved energy absorption and no delamination, whereas the specimens with 9.4 wt.% nanoclay indicated maximum energy absorption with increased delaminated area which is probably due to poor dispersion of nanoparticles and/or agglomeration, which acts as stress concentration areas [3]. High speed impact tests were carried out by Esfahani et al. [3] for glass fibers samples filled with 1.5 and 3 wt.%. The damage assessments of impact area for all specimens showed delamination as being dominant failure mechanism.

6. X-ray results and discussion

The ISOVOLT Titan E stationary X-ray machine (technical data are shown in Table 6) was used to characterize the changes in the internal structure of the different Kevlar laminated composites after impact. This X-ray technique relies on the differential absorption coefficient being directly related to material density and a function of the atomic number or scattering of X-ray photons as they pass through a material. Photos of the front and side views were taken for each of the tested Kevlar composite control sample group DW2 and the NC groups DW3 and DW4. The X-ray films then were exposed to strong illumination source to reveal the

Table 6

The ISOVOLT Titan E stationary X-ray machine technical data.

Parameters	Values
Max. output voltage	160 kV
Max. output current	45 mA
Max. output power	4.5 kW, limited by tube specification
High voltage ripple	5 V/mA (with high voltage cable 10 m), 40 kHz
Insulation	Oil
Housing dimensions (cathode) ($W \times D \times H$)	350 × 870 × 850 mm (13.8" × 34.3" × 33.5")
Weight (cathode)	189 kg (417 lbs), including power module
Weight	4.9 kg (10.8 lbs) including desk housing
Connected loads	
Power connection	1 N PE 230 V ± 10% 50/60 Hz 3 N PE 400/ 230 V ± 10%, 50/60 Hz, 3-phase, grounded neutral TN-S or TN-C-S mains (star connected system, optional 3-phase isolation transformer)
Grounding	Separate grounding for X-ray tube and high voltage generator (minimum 6 mm ²)
Mains fuses	63 A (1 N PE) or 25 A (3 N PE) time-delay fuses, customer-supplied
Operating temperature range	0 °C to +40 °C
Storage temperature range	−30 °C to +70 °C

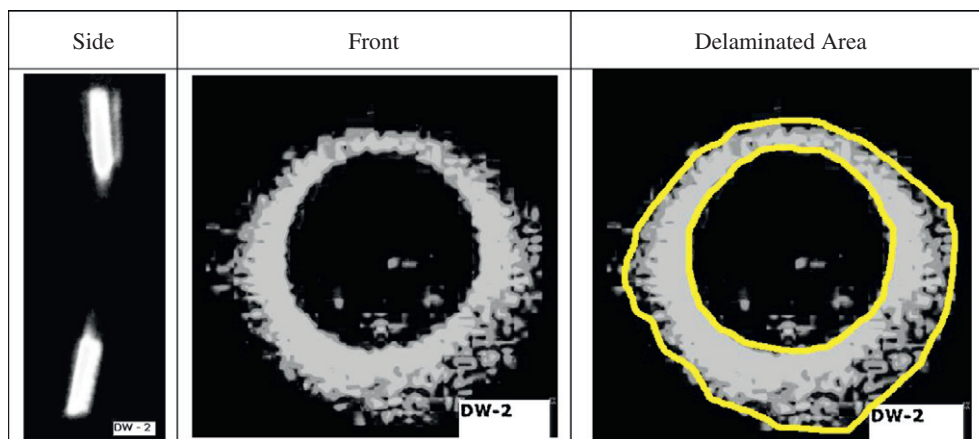
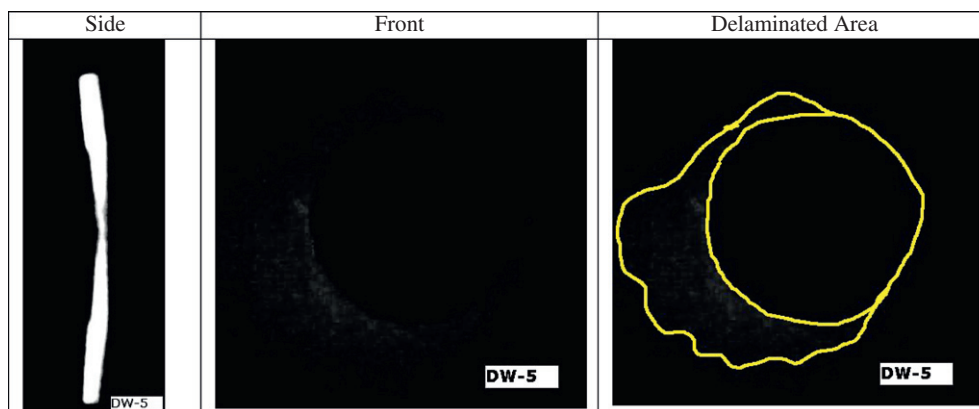
image details, then photos were captured using 10 mega pixels digital camera.

The photos of the X-ray results are shown in Figs. 2–4. These photos show variation in light intensity which is due to the

delaminated areas. The ability of X-ray to penetrate empty places (delaminated) is higher than it is ability in solid regions (non-delaminated). The bright areas represent the delaminated areas while the dark areas represent the remained bonded areas. One can notice considerable match between the front and side view for each sample.

Image processing software was used to modify the contrast and brightness of each photo. This allowed easily prediction of the border line around the delaminated areas as seen in Figs. 2–4. The delaminated percentage of composite areas were calculated and the results were summarized in Fig. 5. It can be observed that the NC composite with 4.30% has the least delamination percentage.

As obvious from Fig. 5, the control samples show high tendency to delaminate (37% of the sample area was delaminated). Fig. 2 demonstrates that the delamination of the control sample started at the circular impacted disk circumference and extended to one of the sample corners. As is clear from Figs. 3 and 5, nanoclay composite with 4.3 wt.% (DW5) played an effective rule in delamination resistance since the delaminated area is reduced to only 22%. The increase in nanoclay percentage to 9.4 wt.% contrary affects the results and almost complete delamination occurs with 71% of the total area was deboned as seen in Figs. 4 and 5. The results revealed enhancement in delamination resistance is achieved at low percentages of additive, while the high additive percentage aggravated the delamination. The nanoclay with low percentage (4.3 wt.%) had shown the best results in delamination resistance.

**Fig. 2.** X-ray results for an impacted control sample (DW2).**Fig. 3.** X-ray results for an impacted of composite containing 4.3 wt.% of NC (DW5).

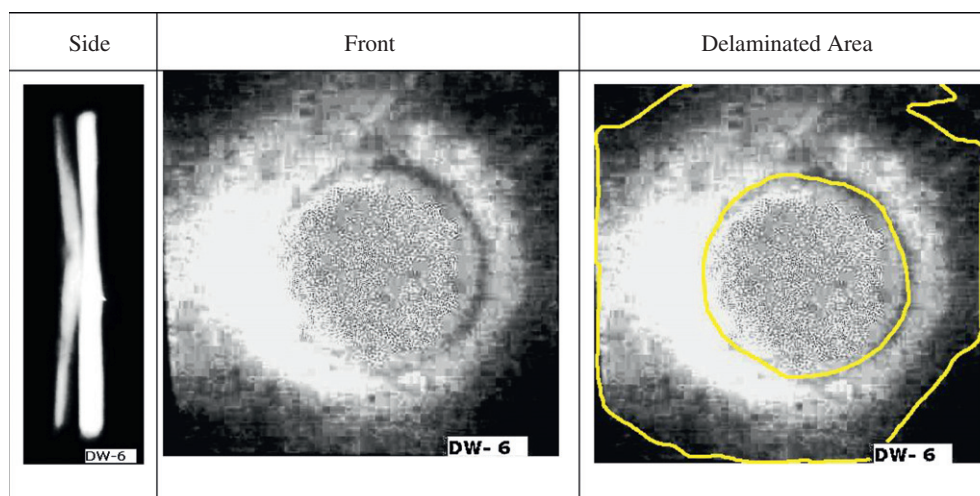


Fig. 4. X-ray results for impacted composite containing 9.4 wt.% of NC (DW6).

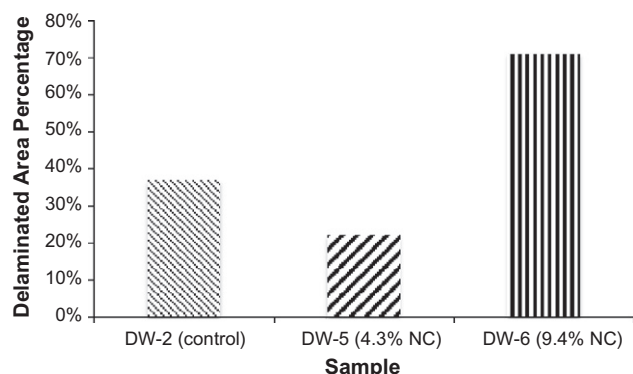


Fig. 5. Percentage of delaminated area of composites with different additives (from X-ray images).

7. Conclusions

Woven Kevlar laminated composites with nanoclay particles additions were tested at different nanoclay percentages. The results were compared to control specimen composed of Kevlar plies only. A vertical drop-weight experimental setup was utilized to test the prepared sample laminates. The impact tests revealed that:

- Enhancement in impact resistance due to nanoclay addition. Control samples were completely penetrated by the impactor at 250 J energy level, whereas the 4.3 wt.% samples showed partial penetration (5 plies only). The 9.4 wt.% samples showed higher impact resistance where the partial penetration went through 2 plies only.
- The high percentage of nanoclay aggravated the composite delamination. X-ray images showed that the 9.4 wt.% samples delaminated area percentage is approximately 71%.
- The nanoclay with low percentage (4.3 wt.%) had shown the best results in delamination resistance.

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